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IS 1448-95 (1980): Methods of test for petroleum and its products, Part 95: Determination of demulsification number of lubricating oils [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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IS : 1448 [P : 95] - 1980
Adopted : 12 March 1980
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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 95]

DETERMINATION OF DEMULSIFICATION NUMBER OF LUBRICATING OILS

(First Reprint JULY 1992)
UDC 621·892 : 541·18·051·3

Adapted from the Institute of Petroleum, UK, Standard No. IP 19/76

1. SCOPE

1.1 This method gives a measure of the ability of the oil to separate from an emulsion. It is commonly applied to turbine oils, but it may be used for other lubricating oils. The test is commonly applied to used turbine oils but since it is sensitive to ageing and contamination of the oil, precision will be lower than that stated.

1.1.1 Oils containing inhibitors may give much higher results than the corresponding uninhibited oils and the precision may be less satisfactory.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definition shall apply.

2.1 Demulsification Number — The number of seconds required for an oil to separate when it is emulsified and separated under specified conditions.

3. OUTLINE OF THE METHOD

3.1 Twenty milliliters of oil is emulsified with steam at about 90°C. The emulsion is then placed in a bath at about 94°C and the time for 20 ml of oil to separate is recorded.

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4. APPARATUS

4.1 The apparatus consists of the following parts and is as shown in Fig. 1.

4.1.1 *Steam Generator* — of metal or glass, and if of metal, fitted with a water-gauge.

4.1.2 *Separating and Emulsifying Baths* — of glass, and provided with wooden or metal covers to hold the oil container and thermometers vertically in the bath.

4.1.3 *Heat Sources* — a suitable burner or electric hot-plate for the steam generator and any convenient means, such as an auxiliary steam line, for the separating bath.

4.1.4 *Oil Container*

4.1.5 *Steam Piping*

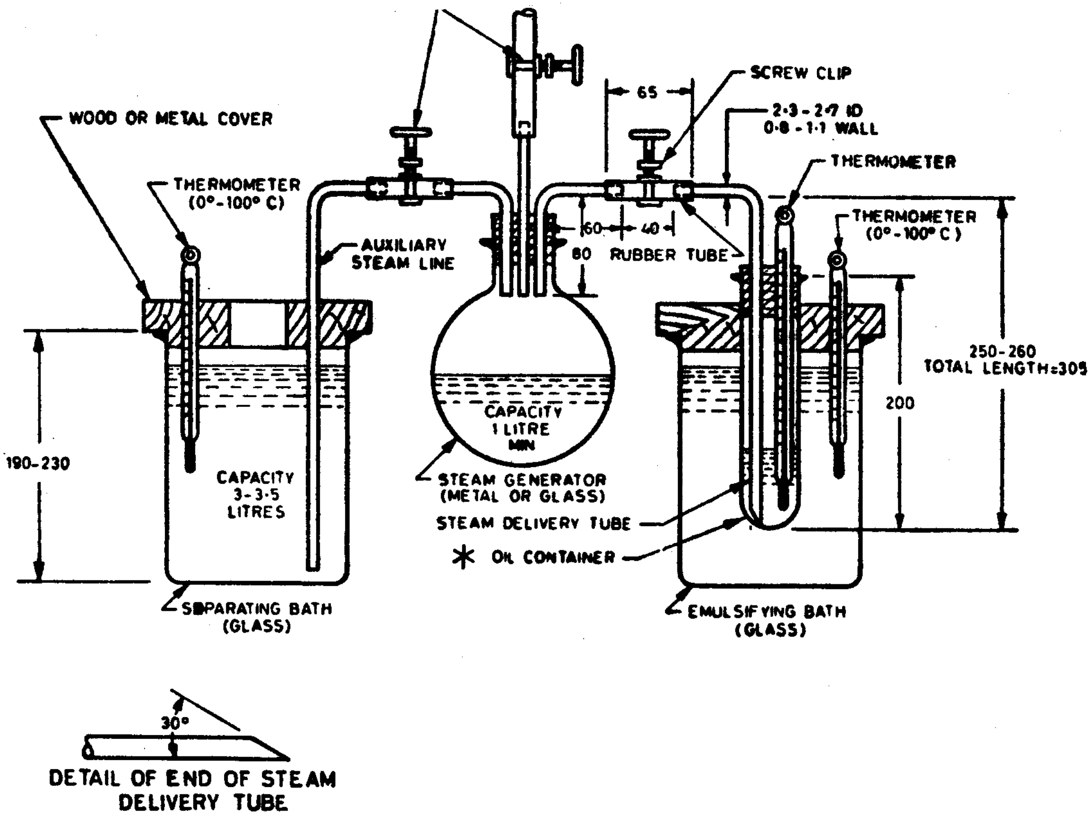
4.1.6 *Thermometers for the Separating and Emulsifying Baths* — of suitable range, that is, 0 to 100°C.

4.1.7 *Thermometer for Oil Container* — conforming to the following requirements:

Range	−1°C to +105°C
Graduation at each	0·5°C
Immersion	Total
Stem diameter	5·5 to 8·0 mm
Bulb shape	Cylindrical
Bulb diameter	Not greater than stem
Length of graduated portion, *Min*	170 mm
Longer lines at each	1°C and 5°C
Figured at each	5°C
Expansion chamber	Required
Top finish	Ring
Scale error, *Max*	± 0·5°C

5. PRECAUTIONS

5.1 The results obtained by this method are readily affected by the presence of traces of impurities in the sample and in consequence, it is most important to avoid contamination of the sample. Take precautions to exclude light from the sample until it can be tested.



*Oil Container of 23 mm Internal Diameter, 0·8 to 1·4 mm Wall Thickness, Graduated from 10 to 50 ml, each 5 ml Line Encircling the Tube. Distance from 10 ml to 50 ml line shall be 98 ± 5 mm.

All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF DEMULSIFICATION NUMBER

6. PREPARATION OF APPARATUS

6.1 Fill the steam generator half-full of water, adding some broken pumice stone for the promotion of even ebullition, and assemble the apparatus as shown in Fig. 1. Take care to exclude from the steam generator any foreign materials which would cause contamination of the steam, and make sure that the steam-piping is clean. Fill each bath with 3 000 ± 60 ml of water.

6.2 Boil the water in the steam generator, adjust the temperature of the water in the emulsifying bath to 19 to 26°C and in the separating bath to 93 to 95°C.

7. PROCEDURE

7.1 Carry out the test in duplicate.

7.2 Ensure that all parts of the apparatus which come into contact with the sample are chemically clean, since this test is very susceptible to contaminants. Measure 20 ml of the sample into the oil container and place the latter in the holder of the emulsifying bath. Place a cork with two openings, with the thermometer in one, in the mouth of the oil container, adjusting the thermometer so that the bottom of the bulb is 20 to 25 mm from the bottom of the tube.

7.3 Pass steam through the steam-delivery tube until the condensate disappears, and then disconnect it from the rubber tubing and insert it through the second opening in the cork until the end of the tube touches the centre of the bottom of the oil container. The fit between the steam-delivery tube and the cork should be loose. Re-connect the rubber tubing to the steam-delivery tube and admit steam at such a rate that the temperature of the oil, as indicated by the thermometer in the oil container, rises to 88 to 91°C. The usual time for the temperature of the oil to reach this point is 45 to 75 seconds depending on its nature. Control the temperature by regulating the screw clips on steam pipe and steam exhaust, making sure that the steam supply is sufficient at all times to cause a generous discharge from the exhaust. Continue passing steam until the volume of the condensed water and the oil in the oil container is 40 ± 3 ml, making appropriate allowance for the volume of steam, the steam-delivery tube and the thermometer; this volume is approximately 12 to 15 ml. The time for this operation should be 4 to 6·5 minutes. If the time is less than 4 minutes, reject the test as the steam is probably wet, or the steam-delivery tube was completely steamed out.

7.4 Disconnect the steam-delivery tube from the rubber tubing and immediately start a stop-watch. Without delay, transfer the oil container to the separating bath; it is extremely important that the separating bath be maintained at 93 to 95°C. Remove the thermometer and steam-delivery tube from the oil container (*see* Note). After every 30 seconds record the volume of the separated oil layer, making no differentiation between the clear and turbid oil. Where the interface between more or less clear oil and the emulsion is not a clear, straight, horizontal line, estimate the position of such a line to the nearest 0·5 ml, mid-way through the more or less clear oil bubbles. Consider any lacy emulsion as part of the water layer. Record the time in seconds at the end of the 30-second interval during which 20 ml of oil separates. If 20 ml of oil has not separated in 20 minutes, discontinue the test and record the volume of oil separated.

NOTE — The oil container shall not be withdrawn from the separating bath during the period of examination.

8. CALCULATION AND REPORTING

8.1 Calculate the mean of the results of the duplicate determinations. If these results differ from the mean by more than 15 seconds or by more than 10 percent of the mean, if this is greater, make a third test.

8.2 Report the mean result to the nearest 15 seconds as the demulsification number. If 20 ml of oil has not separated in 20 minutes, report the demulsification number as 1 200 + (number of millilitre separated).

9. PRECISION

9.1 Results of duplicate tests should not differ by more than the following amounts:

	Repeatability	*Reproducibility*
Unused oil	30 seconds or 20 percent of mean, whichever is greater	60 seconds or 30 percent of mean whichever is greater